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DEVELOPMENT OF HIGH TEMPERATURE  
MULTIPROPERTY APPARATUS

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# **DEVELOPMENT OF HIGH TEMPERATURE MULTIPROPERTY APPARATUS**

*R. E. TAYLOR*

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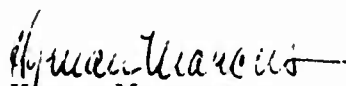


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## FOREWORD

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## ABSTRACT

The development of the multiproperty apparatus to include high temperature determinations of specific heat, enthalpy and thermal diffusivity in addition to the properties already measured is described. The latter properties include thermal conductivity, electrical resistivity, Lorenz ratio, total hemispherical emittance, spectral emittance at 0.65 microns and Thomson coefficient. The specific heat determination involves small transients from an established steady-state temperature. The accuracy and reproducibility of the specific heat determination is controlled by the accuracy obtained in measuring the rate of change of temperature. When photographing an oscilloscopic trace of an automatic pyrometer output, the inaccuracy was about 8 percent. By using a digital data acquisition system, it is believed that data accurate within 1 percent can be achieved at high temperatures.

## TABLE OF CONTENTS

|  | Page |
|--|------|
| I. INTRODUCTION                                | 1    |
| II. BACKGROUND ON SPECIFIC HEAT DETERMINATIONS | 10   |
| III. EXPERIMENTAL APPARATUS AND PROCEDURES     | 12   |
| IV. EXPERIMENTAL RESULTS AND DISCUSSION        | 24   |
| V. SUMMARY AND CONCLUSIONS                     | 28   |
| VI. REFERENCES                                 | 28   |

## LIST OF ILLUSTRATIONS

### Figure

|   |    |
|---|----|
| 1. Electrical Resistivity of Tungsten                         | 3  |
| 2. Total Hemispherical Emittance of Tungsten                  | 5  |
| 3. Thermal Conductivity of Tungsten                           | 7  |
| 4. Cooling Rates Resulting from Incremental Change in Current | 13 |
| 5. View of Multiproperty Apparatus                            | 15 |
| 6. Response of Power Supplies to Step Changes                 | 17 |
| 7. Typical Temperature Versus Time Recording                  | 21 |
| 8. Specific Heat of Tantalum                                  | 25 |

## LIST OF TABLES

### Table

|  |    |
|--|----|
| I. Magnitudes of Conduction, Heating, Radiation, and Thomson Terms at Selected Positions along a Typical Temperature Profile | 9  |
| II. Specific Heat of Tantalum  | 10 |
| III. Typical Calibration of Electronic Output  | 20 |
| IV. Specific Heat Calculation  | 23 |

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## NOMENCLATURE

| Symbol            | Definition  |
|-------------------|---|
| $A$               | Cross-sectional area perpendicular to Z-axis        |
| $BT$              | Brightness temperature                              |
| $C_p$             | Specific heat                                       |
| $I$               | Electric current                                    |
| $I_\infty$        | Electric current through infinitely long sample     |
| $J$               | Joules  |
| $P$               | Circumference of sample                             |
| $T$               | Temperature of the sample at any location $Z$       |
| $\dot{T}$         | Rate of change of temperature                       |
| $TT$              | True temperature                                    |
| $T_o$             | Ambient temperature                                 |
| $T_\infty$        | Temperature at center of infinitely long sample     |
| $V$               | Voltage drop  |
| $W$               | Watts   |
| $Z$               | Length coordinate along longitudinal axis of sample |
| $\alpha$          | Total hemispherical absorptance                     |
| $\epsilon_H$      | Total hemispherical emittance                       |
| $\epsilon_{0.65}$ | Spectral emittance at 0.65 microns                  |
| $\delta$          | Density   |
| $\lambda$         | Thermal conductivity                                |
| $\mu$             | Thomson coefficient                                 |
| $\rho$            | Electrical resistivity                              |
| $\sigma$          | Stefan-Boltzmann constant                           |

## I. INTRODUCTION

There are two common methods of measuring the thermal conductivity of solids at high temperatures. These are the radial heat flow method in which a calibrated heater or heat sink is inserted in the center of a tubular sample and the radial temperature gradient determined, and the direct heating method in which a long thin sample is clamped between two electrodes and heated by passing regulated current through it while determining the longitudinal temperature gradient. The direct heating method is simpler experimentally but more complex mathematically.

For example the differential equation describing energy transport in a long thin rod at steady state heated electrically in vacuo and free to radiate to its enclosure is

$$\frac{d}{dZ} \left( \lambda \frac{dT}{dZ} \right) + \frac{I^2 \rho}{A^2} - \frac{P \epsilon_H \sigma}{A} (T^4 - T_0^4) - \mu \frac{1}{A} \frac{dT}{dZ} = 0 \quad \text{Eq. (1)}$$

where  $I$  is the DC current,  $A$  is the cross-sectional area,  $P$  is the circumference,  $\sigma$  is the Stefan-Boltzmann constant,  $T$  is the temperature at any longitudinal position  $Z$ ,  $T_0$  is the temperature of the enclosure,  $\lambda$  is the thermal conductivity,  $\rho$  is the electrical resistivity,  $\epsilon_H$  is the total hemispherical emittance and  $\mu$  is the Thomson coefficient. There are several important things to note in Eq. (1). First,  $\lambda$ ,  $\rho$ ,  $\epsilon_H$ , and  $\mu$  are functions of temperature (and therefore of position). There is no known closed solution for  $\lambda$  even for the relatively simple case in which  $\mu = 0$  and the temperature dependencies of  $\lambda$ ,  $\rho$  and  $\epsilon_H$  are negligible. The experimentalist measures  $T$  as a function of  $Z$ , whereas Eq. (1) contains  $dT/dZ$  and  $d^2T/dZ^2$  in addition to powers of  $T$ . Therefore, it is necessary to employ very restrictive mathematical approximations in order to linearize Eq. (1), use numerical integration techniques or differentiate an appropriate expression for  $T(Z)$  to obtain  $dT/dZ$  and  $d^2T/dZ^2$  in order to obtain  $\lambda$  from the experimental data. The various variants have been described in several papers <sup>(1,2,3)</sup>. Taylor and coworkers <sup>(4,5,6,7,8)</sup> have developed several analytical schemes which permit the calculation of  $\lambda$  from the experimental data without the use of restrictive mathematical approximations. Using these techniques they have obtained very accurate data for  $\lambda$ ,  $\rho$  and  $\epsilon_H$  to high temperatures <sup>(2,5,8,9)</sup>. For example, portions of the  $\rho$  and  $\epsilon_H$  curves for arc-cast tungsten <sup>(5)</sup> are shown in Figures 1 and 2 while the  $\lambda$  results <sup>(5)</sup> are shown in Figure 3.

Taylor et al. (2, 5) showed the necessity of measuring  $\rho$ ,  $\epsilon_H$  and  $\lambda$  on the same sample in the same apparatus. Basically the reason for this is that small uncertainties in  $\rho$  or  $\epsilon_H$  cause a large uncertainty in  $\lambda$  because the second and third terms of Eq. (1) are of nearly equal magnitude and the fourth term is relatively small. Thus the magnitude of the term containing  $\lambda$  is essentially the difference between the magnitudes of nearly equal terms. This is illustrated in Table I in which the magnitudes of the various terms at selected positions along a typical profile are computed. At the position  $Z = 22.642$  cm, it is seen that the Joulean heating term  $I^2\rho/A^2$  is 677.165 watts/cm<sup>3</sup>, the radiation term is 640.662 watts/cm<sup>3</sup> and the Thomson term is 1.021 watts/cm<sup>3</sup>. The term containing  $\lambda$  is only 35.481 watts/cm<sup>3</sup> and thus a small error in  $\rho$  or  $\epsilon_H$  has a large effect on this term. Because of this situation, the successful employment of the direct heating method involves multiproperty determinations.

While the mathematical techniques have not been optimized as yet for determining  $\mu$ , it appears feasible to do so and Taylor and coworkers are pursuing this objective (2). Also the spectral emittance at 0.65 microns is readily determined from the experimental data generated during a direct heating experiment. Since  $\lambda$  and  $\rho$  are determined on the same specimen, the Lorenz function can also be calculated from the data. Thus the direct heating method has been used to measure six properties simultaneously on the same sample.

Because of the ideal sample geometry, it appears feasible to measure at least seven additional thermophysical properties on the same sample with accuracies comparable to those obtainable under optimum conditions for each property. These additional properties are thermal expansion, specific heat, enthalpy, thermal diffusivity, Seebeck coefficient, Peltier coefficient, and Richardson coefficient. The present project has as its goal the extension of the direct heating technique to include specific heat determinations. Since the enthalpy can be obtained by merely integrating the specific heat results, it would be calculable from the specific heat data. Similarly, because the thermal conductivity would be determined on the same specimen, measuring the specific heat would permit the calculation of thermal diffusivity, provided the density is known.

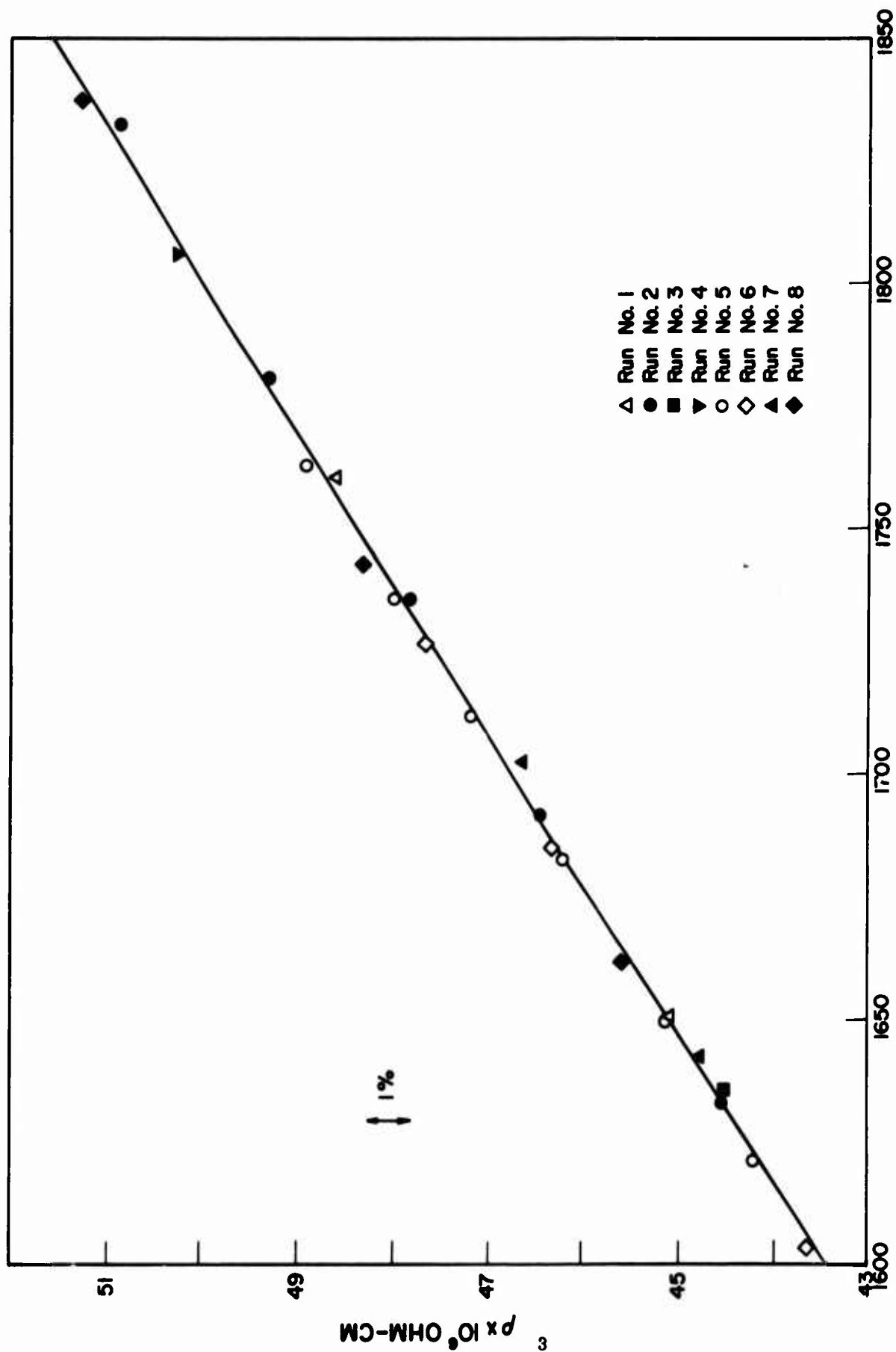


FIGURE 1. ELECTRICAL RESISTIVITY OF TUNGSTEN

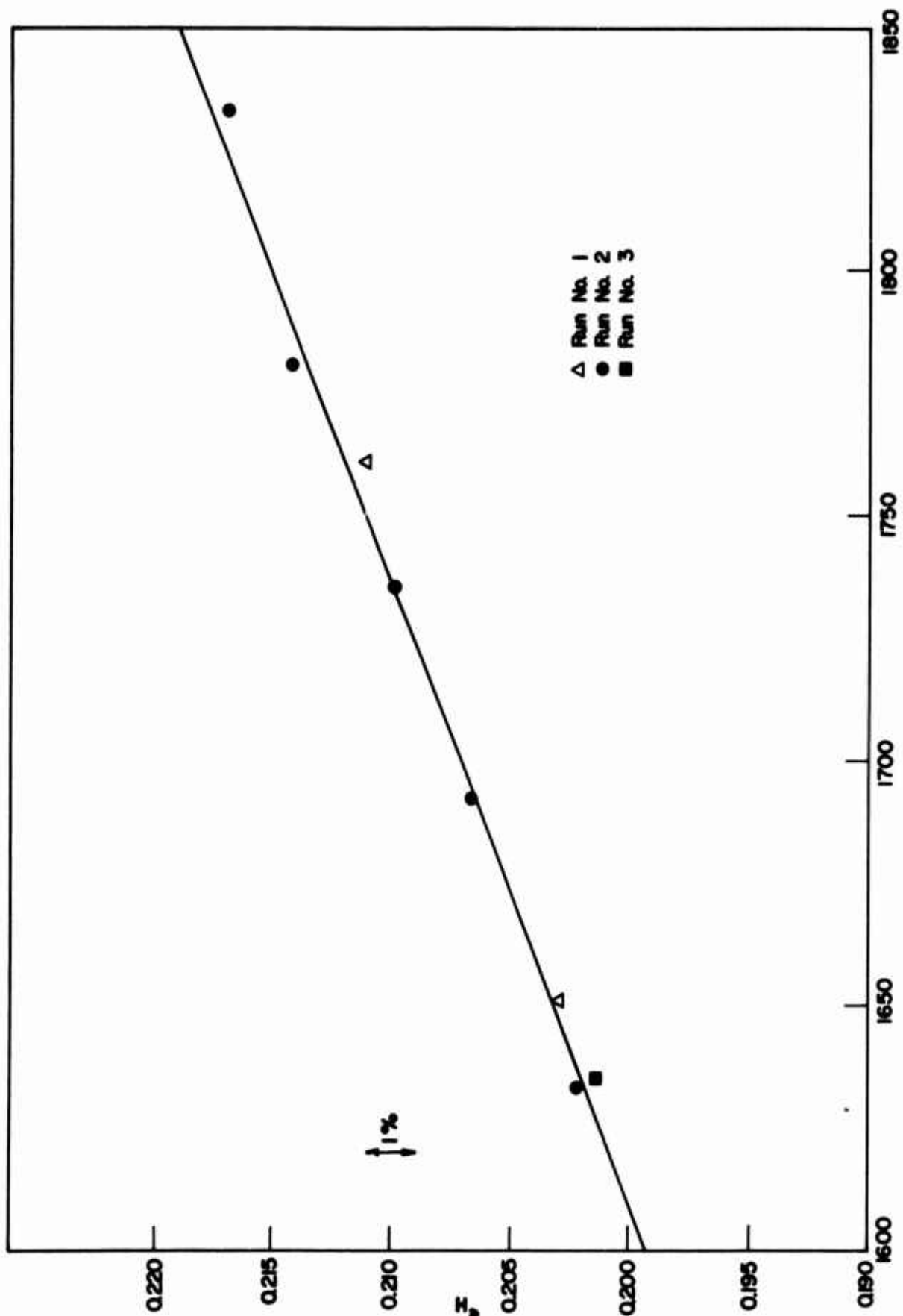


FIGURE 2. TOTAL HEMISPHERICAL EMITTANCE OF TUNGSTEN

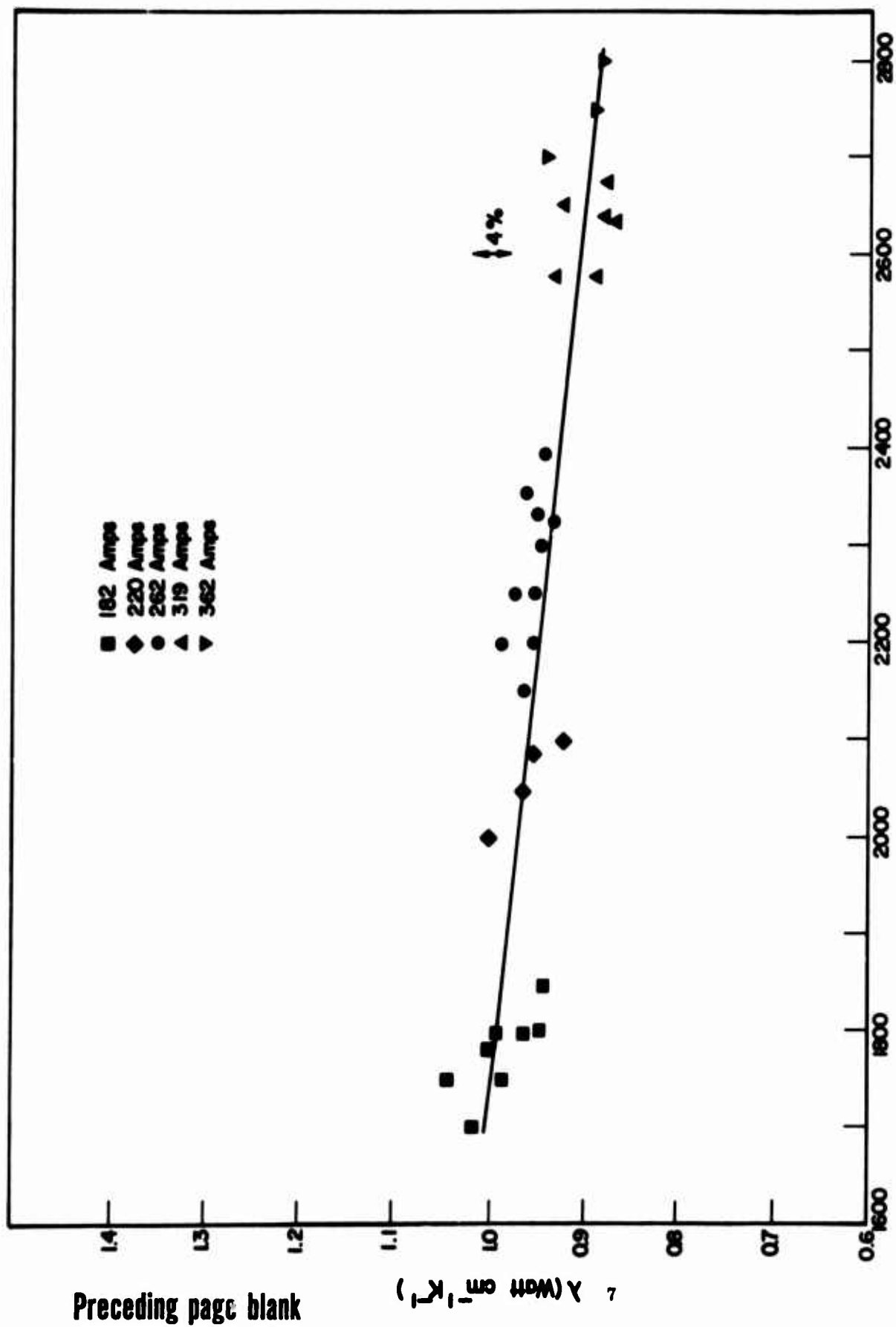


FIGURE 3. THERMAL CONDUCTIVITY OF TUNGSTEN

TABLE I

MAGNITUDES OF CONDUCTION, HEATING, RADIATION, AND THOMSON  
TERMS AT SELECTED POSITIONS ALONG A TYPICAL TEMPERATURE PROFILE

| Z<br>(cm) | T<br>(K) | dT/dZ<br>(K/sec) | Conduction<br>(W/cm <sup>2</sup> ) | Heating<br>(W/cm <sup>2</sup> ) | Radiation<br>(W/cm <sup>2</sup> ) | Thomson<br>(W/cm <sup>2</sup> ) |
|-----------|----------|------------------|------------------------------------|---------------------------------|-----------------------------------|---------------------------------|
| 20.2400   | 2181.62  | 255.351          | 192.085                            | 597.135                         | 389.271                           | 15.779                          |
| 20.3901   | 2217.66  | 225.173          | 174.380                            | 608.813                         | 420.521                           | 13.911                          |
| 20.5403   | 2249.38  | 197.859          | 157.334                            | 619.115                         | 449.558                           | 12.224                          |
| 20.6904   | 2277.21  | 173.280          | 141.202                            | 628.181                         | 476.273                           | 10.705                          |
| 20.8406   | 2301.55  | 151.271          | 126.168                            | 636.128                         | 500.615                           | 9.345                           |
| 20.9907   | 2322.76  | 131.640          | 112.341                            | 643.071                         | 522.598                           | 8.132                           |
| 21.1409   | 2341.19  | 114.184          | 99.771                             | 649.115                         | 542.291                           | 7.054                           |
| 21.2910   | 2357.15  | 98.694           | 88.465                             | 654.358                         | 559.796                           | 6.097                           |
| 21.4412   | 2370.91  | 84.964           | 78.397                             | 658.888                         | 575.242                           | 5.249                           |
| 21.5913   | 2382.74  | 72.794           | 69.520                             | 662.783                         | 588.767                           | 4.497                           |
| 21.8916   | 2401.41  | 52.379           | 55.091                             | 668.945                         | 610.618                           | 3.236                           |
| 22.0418   | 2408.62  | 43.784           | 49.406                             | 671.326                         | 619.215                           | 2.705                           |
| 22.1919   | 2414.61  | 36.047           | 44.646                             | 673.303                         | 626.421                           | 2.227                           |
| 22.3421   | 2419.48  | 29.020           | 40.783                             | 674.916                         | 632.341                           | 1.792                           |
| 22.4922   | 2423.35  | 22.563           | 37.738                             | 676.195                         | 637.064                           | 1.393                           |
| 22.6424   | 2426.28  | 16.542           | 35.481                             | 677.165                         | 640.662                           | 1.021                           |
| 22.7925   | 2428.33  | 10.830           | 33.986                             | 677.844                         | 643.190                           | 0.669                           |
| 22.9427   | 2429.54  | 5.303            | 33.234                             | 678.244                         | 644.683                           | 0.327                           |
| 23.0928   | 2429.93  | - 0.161          | 33.224                             | 678.372                         | 645.159                           | -0.011                          |
| 23.2430   | 2429.49  | - 5.684          | 33.962                             | 678.226                         | 644.616                           | -0.352                          |

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## II. BACKGROUND ON SPECIFIC HEAT DETERMINATIONS

The direct determination of specific heat ( $C_p$ ) at high temperatures has been made using pulse heating<sup>(10, 11)</sup> or modulating current techniques<sup>(13, 14)</sup>. The specific heat has also been determined at high temperatures by measuring the enthalpy as a function of temperature by drop-calorimetry and differentiating. The results of these various approaches are often in good agreement from room temperature to temperatures about 2/3 of the melting point but often diverge markedly as the temperature approaches the melting point<sup>(10, 12)</sup>. For example, compare the data of Hoch and Johnston<sup>(16)</sup> (drop calorimetry) and Taylor and Finch<sup>(10)</sup> (pulse heating) for tantalum given in Table II. The results obtained by the modulation method by Lowenthal<sup>(13)</sup> agree with the Taylor and Finch results, whereas the modulation results of Kraftmahker<sup>(14)</sup> agree with Hoch and Johnston.

TABLE II  
SPECIFIC HEAT OF TANTALUM (J/gm K)

| T (K) | Hoch and Johnston <sup>(16)</sup> | Taylor and Finch <sup>(10)</sup> | Rasor and McClelland <sup>(11)</sup> | Lowenthal <sup>(13)</sup> | Kraftmahker <sup>(14)</sup> |
|-------|-----------------------------------|----------------------------------|--------------------------------------|---------------------------|-----------------------------|
| 2000  | 0.1658                            | 0.1713                           | 0.1746                               | 0.1710                    | 0.1660                      |
| 2200  | 0.1683                            | 0.1754                           | 0.1807                               | 0.1764                    | 0.1692                      |
| 2400  | 0.1709                            | 0.1808                           | 0.1884                               | 0.1825                    | 0.1723                      |
| 2600  | 0.1732                            | 0.1896                           | 0.1979                               | -                         | 0.1754                      |
| 2800  | 0.1756                            | 0.2026                           | 0.2111                               | -                         | 0.1786                      |
| 3000  | -                                 | 0.2205                           | 0.2328                               | -                         | -                           |

The dependence of the specific heat on temperature as determined from enthalpy data is critically dependent on the form of the polynomial chosen to represent the enthalpy data. Also, the results from drop calorimetry are subject to question since the reference state may not be re-attained during the quenching operation. Pulse heating experiments require the accurate determination of the rate of temperature change during transients but with recent improvements in instrumentation this need not be a major obstacle. Thus pulse heating techniques are superior to drop calorimeter techniques for high temperature specific heat determinations for electrical conductors.

The direct heating experiment is clearly adaptable to specific heat determinations using pulse heating or cooling. The governing equation then becomes



$$\frac{d}{dz} \left( \lambda \frac{dT}{dz} \right) + \frac{I^2 \rho}{A^2} - \frac{P \epsilon_H \sigma}{A} (T^4 - T_o^4) - \mu \frac{1}{A} \frac{dT}{dz} = C_p \delta \dot{T} \quad \text{Eq. (2)}$$

where  $\delta$  is the density and  $\dot{T}$  is the rate of temperature change. For long rods  $dT/dz = 0$  and Eq. (2) becomes

$$\frac{I^2 \rho}{A^2} - \frac{P \epsilon_H \sigma}{A} (T^4 - T_o^4) = C_p \delta \dot{T} \quad \text{Eq. (3)}$$

Previous users of this general method at high temperatures have used wire samples <sup>(10)</sup>, thin tubes <sup>(12)</sup> or an auxiliary external heater <sup>(11)</sup>. The use of an auxiliary heater permits relatively small transients from an arbitrary base temperature. However, the use of the heater greatly increases the apparatus complexity and the radiation loss problem. In this case Eq. (3) becomes

$$\frac{I^2 \rho}{A^2} - \frac{P \sigma \epsilon_H}{A} (T^4 - \frac{\alpha}{\epsilon_H} T_o^4) = C_p \delta \dot{T} \quad \text{Eq. (4)}$$

where  $\alpha$  is the total hemispherical absorptance of the sample and the term  $\alpha T_o^4 / \epsilon_H$  must be determined accurately. When a heater is not used, pulse heating to high temperature may use large heating rates and Eq. (3) becomes

$$\frac{I^2 \rho}{A^2} = C_p \delta \dot{T} \quad \text{Eq. (5)}$$

when the radiation losses are made negligible. However, rapid pulsing from room temperature causes large temperature transients and possible changes in the sample. Therefore, for use with the multiproperty apparatus it was decided to use incremental changes in the power input to achieve transients. This approach does not require significant modifications to the experimental apparatus except for the data collection system and does not put undue stress upon the sample nor does it involve quenching.

One may, of course, increase or decrease the rate of power input and thus observe either cooling or heating of the specimen. Rates of cooling that can be achieved from a 1/8 inch diameter specimen of tantalum by incrementally decreasing the current are plotted in Figure 4. From this figure it can be seen that initial cooling rates at 2000 K are 15 C/sec for a 10 percent change in current and increase to 93 C/sec for a 100 percent decrease in power. These rates are very modest compared to the  $10^3$  to  $10^5$  C/sec used by the researchers using pulse heating at high temperatures without an auxiliary heater and should not cause changes in the sample structure.

### III. EXPERIMENTAL APPARATUS AND PROCEDURES

The basic apparatus for steady-state measurements is described in the literature <sup>(4, 5, 8)</sup>. Briefly it consists of a high vacuum ( $10^{-8}$  torr) chamber equipped with an optical window, regulated power supplies, and a guarded six-dial potentiometer. The sample is clamped between movable electrodes inside the vacuum chamber. Temperature measurements are made using an automatic optical pyrometer mounted on an external platform which can be raised or lowered to view the entire specimen through the optical window.

An overall view of the apparatus is shown in Figure 5. The automatic high vacuum system with bell jar equipped with two rectangular optical windows is shown to the left of center. The automatic optical pyrometer mounted on the platform is located in front of one of these optical windows. The regulated DC power supplies are located in panels near the right side of the picture. The guarded six-dial potentiometer facility is not visible in this view.

In order to perform the transient measurements in which the rate of power is incrementally changed, it was necessary to provide a means of rapidly changing the rate of power input and to provide a means of measuring the rate of temperature change. The output of the power supplied was changed by switching a variable resistor in parallel with the control resistor. By changing the value of the variable resistor it was possible to change the current level over a wide range and thus control the rate of cooling or heating. This switching network is visible on top of one of the regulated power supplies (Figure 5).

It was found that the Kepco power supplies on hand could be decreased from one current setting to another within 250 milliseconds including overshoot when the power supplies were operating in the constant current mode. The level would change to  $1/e$  of its final value within 100 milliseconds. The time response was about half of this value when the current level was increased. The typical responses are shown in Figure 6. Thus by neglecting the data obtained during the first 0.3 seconds, the use of Kepco power supplies was feasible.

Unfortunately, the largest Kepco supply available is capable of delivering only 100 amps. By paralleling two one-hundred amp supplies, it was possible to obtain 200 amps at 8 volts. Using this arrangement it was possible to incrementally change the temperature of the usual size metal samples (0.125 in. dia. by 12 in. long rods) up to 2500 K. By paralleling a third Kepco supply (on hand) and

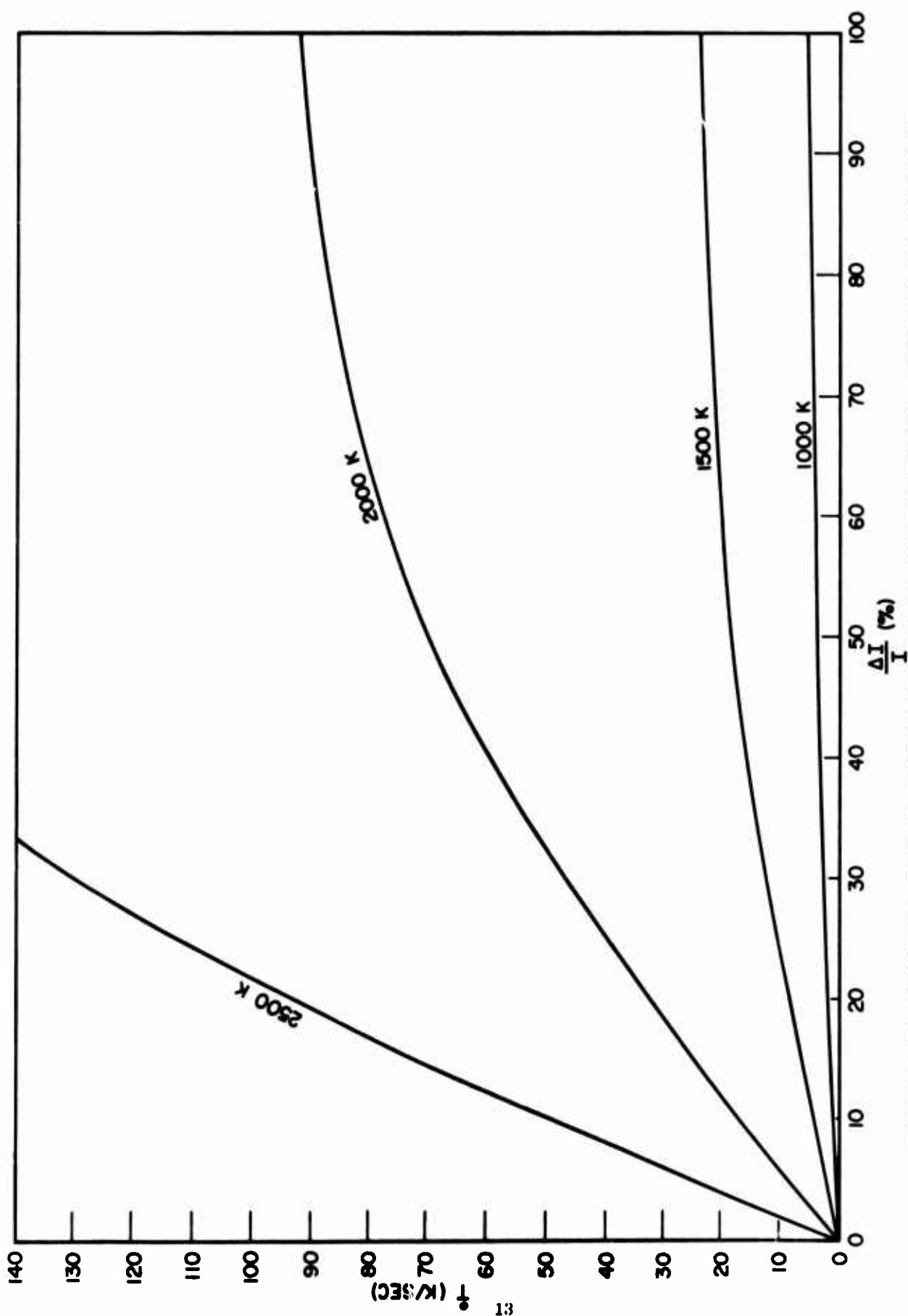


FIGURE 4. COOLING RATES RESULTING FROM INCREMENTAL CHANGE IN CURRENT

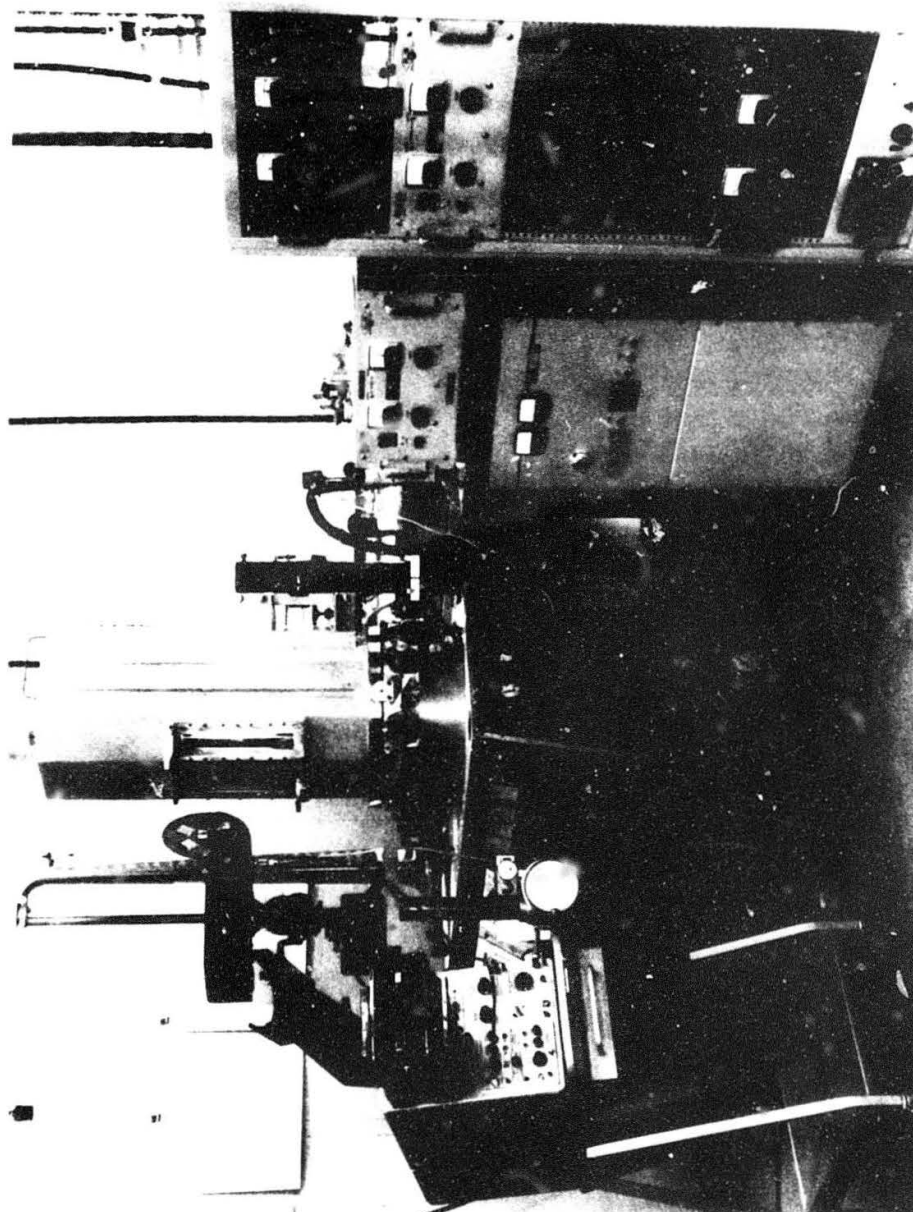


FIGURE 5. VIEW OF MULTIPROPERTY APPARATUS

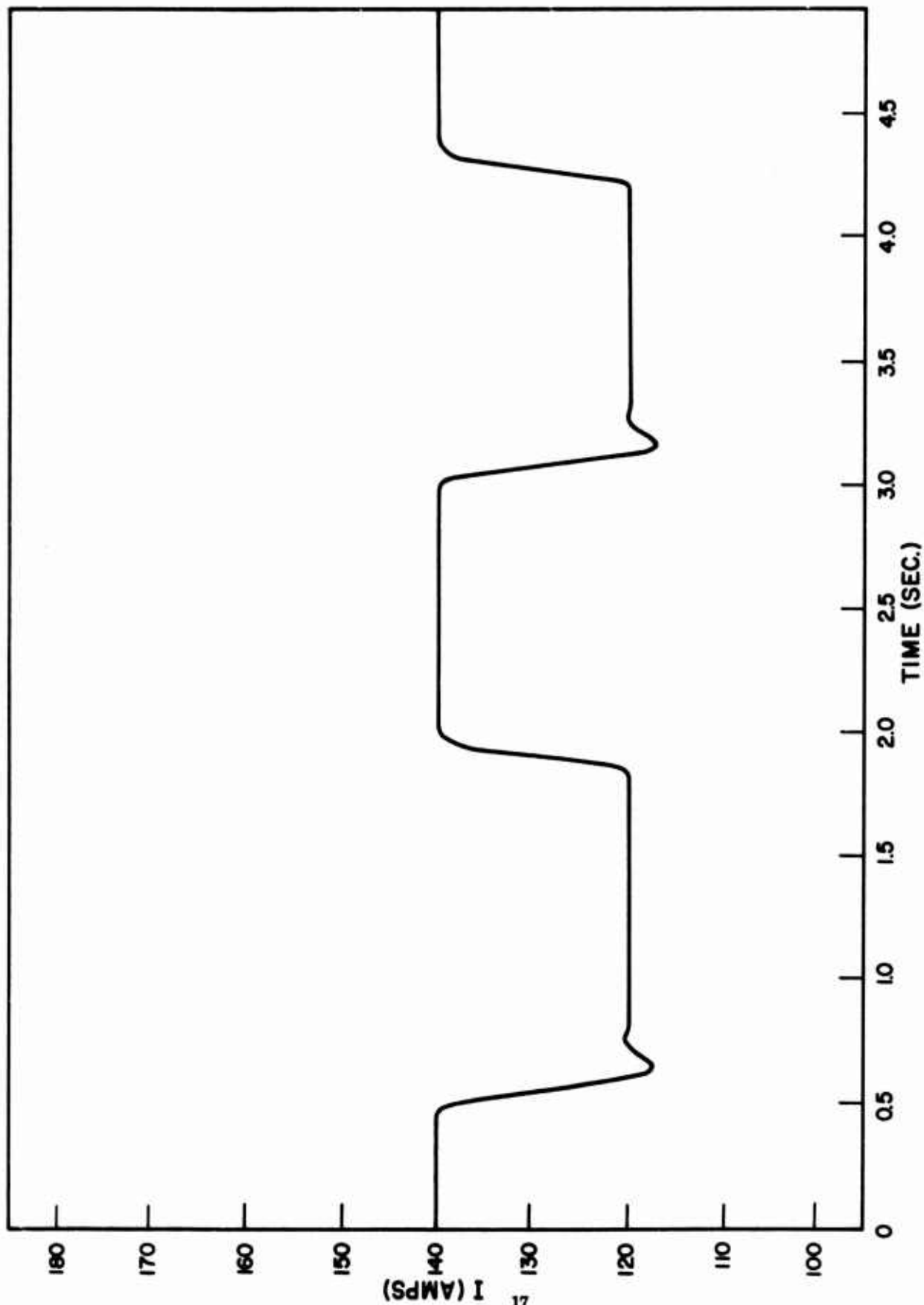


FIGURE 6. RESPONSE OF POWER SUPPLIES TO STEP CHANGES

decreasing the effective sample length to 10 inches, it should be possible to attain 3000 K. Larger electrical conducting cable is required in order to incorporate this modification.

The automatic optical pyrometer used can be operated in the transient mode by operating directly off the photomultiplier tube. However, this signal-to-noise ratio is unsatisfactory and the signal must be filtered. The response time is controlled by the filtering network. Consequently a combination DC amplifier and AC filter was designed and constructed specifically for this transient work. This electronic device is located just below the automatic pyrometer (Figure 5). The output of the filter is fed into an oscilloscope (shown in Figure 5) or into an X-Y recorder (not visible in Figure 5). The sensitivity of the pyrometer-amplifier-filter circuit is controlled by varying the high voltage supplied to the pyrometer photomultiplier and by varying the resistance in the amplifier circuit. Sensitivities from 5 to 50 mV/C were commonly used. Several neutral-density filters were mounted on a rotating wheel on the front of the pyrometer (Figure 5) and were used while adjusting the sensitivity of the electronic output.

After attaining a satisfactory sensitivity, the electronic system was calibrated. This was done either using a recorder or an oscilloscope. First the transient output was adjusted to approximately the top of the scale on the recording device and a trace of this output was recorded. Then the current to the sample, voltage drop across the voltage probes and the pyrometer output (reference mode) were recorded. The pyrometer was switched back to the transient mode and the sample current was changed until the transient output approximated one scale division less on the recording device. A trace was made of this output and the current, voltage and pyrometer output (reference mode) recorded. This procedure was repeated until the last available scale mark on the recording device was used. A typical calibration is shown in Table III. The sample current was then returned to its original value ( $I_{\text{initial}}$ ) and the final current ( $I_f$ ) adjusted to a desired value by temporarily switching in a variable resistor. After the sample reached equilibrium at  $I_{\text{initial}}$ , the current was switched to  $I_f$  and the temperature was recorded as a function of time. Using the same calibration, other values of  $I_f$  were chosen and additional traces were obtained. An oscilloscope recording is illustrated in Figure 7 for the calibration of Table III.

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TABLE III

TYPICAL CALIBRATION OF ELECTRONIC OUTPUT  
(Tantalum Sample)

| Calibration Mark | Sample Current (Amps) | Voltage Drop <sup>†</sup> (Volts) | Pyrometer Output (MV) | Sample Temp. (K) <sup>*</sup> |
|------------------|-----------------------|-----------------------------------|-----------------------|-------------------------------|
| 1                | 117.705               | 0.30166                           | 299.05                | 1906                          |
| 2                | 114.960               | 0.29183                           | 295.45                | 1885                          |
| 3                | 111.947               | 0.28121                           | 291.11                | 1860                          |
| 4                | 108.755               | 0.27009                           | 286.83                | 1837                          |
| 5                | 104.923               | 0.25688                           | 281.61                | 1808                          |
| 6                | 100.320               | 0.24127                           | 275.46                | 1774                          |

<sup>†</sup>  $L = 2.4623 \text{ cm}$ ,  $A = 0.072923 \text{ cm}^2$ ,  $\delta = 16.595 \text{ gm/cm}^3$

<sup>\*</sup> From pyrometer output and emittance and window corrections.

Using the data of Table III and Figure 7 it is possible to calculate specific heat values. The Table III data can be considered to be a series of equilibrium values for an infinitely long sample. Under these conditions

$$\frac{I_{\infty}^2 \rho_{\infty}}{A^2} = P \epsilon_{H\infty} \sigma (T_{\infty}^4 - T_o^4) \quad \text{Eq. (6)}$$

The data from Figure 7 can be considered to be for the nonequilibrium infinitely-long sample (Eq. 3). At any particular instant during the transient Eq. (3) may be written as

$$\frac{I_f^2 \rho}{A^2} - \frac{P \epsilon_{H\infty} \sigma (T_{\infty}^4 - T_o^4)}{A} = C_p \delta \dot{T} \quad \text{Eq. (7)}$$

Substituting Eq. (6) into Eq. (7)

$$\frac{I_f^2 \rho}{A^2} - \frac{I_{\infty}^2 \rho_{\infty}}{A^2} = C_p \delta (\dot{T})_{\infty} \quad \text{Eq. (8)}$$

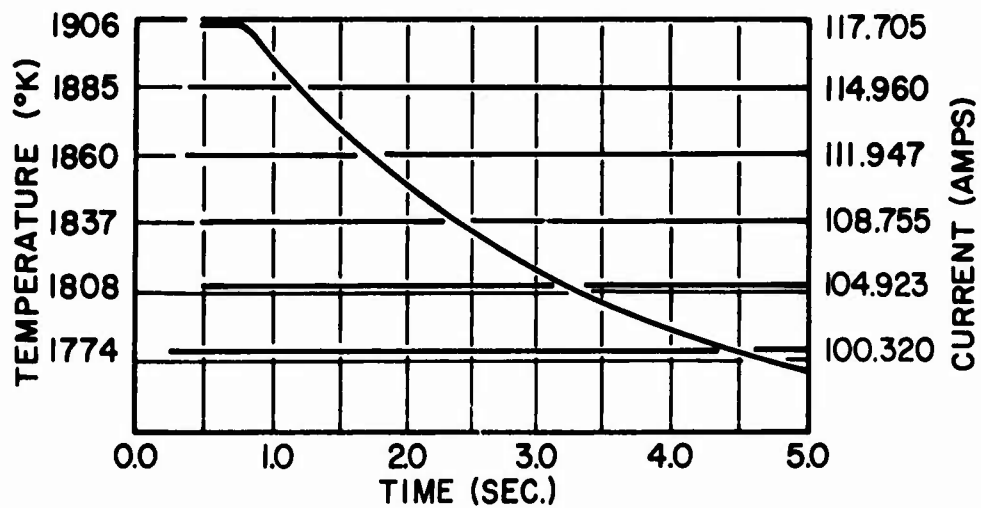


FIGURE 7. TYPICAL TEMPERATURE VERSUS TIME RECORDING



Since at each instant during the transient,  $\rho = \rho_{\infty}$  and  $T = T_{\infty}$

$$C_p = \frac{\rho}{A^2} \frac{(I_f^2 - I_{\infty}^2)}{\delta (\dot{T})_{\infty}} \quad \text{Eq. (9)}$$

From Table III and the sample geometry,  $\rho$  may be calculated

$$\rho = \frac{\Delta E \Delta}{I_{\infty} L} \quad \text{Eq. (10)}$$

The calculations for the data of Figure 7 and Table III are given in Table IV.

All factors except  $(\dot{T})_{\infty}$  entering into Eq. (9) are known to about 5 significant figures (Table IV). Thus the accuracy of the specific heat determination is strictly controlled by the accuracy obtainable for  $(\dot{T})_{\infty}$ . Values for  $(\dot{T})_{\infty}$  are obtained from the slope of the curve at various values of each  $T_{\infty}$  (Figure 7). Using mechanical recorders or oscilloscopes, the usual accuracy is about 5 percent. This may be improved by about a factor of two by very careful and arduous efforts. However, an improvement by an order of magnitude could be achieved using the procedures outlined in the following section.

TABLE IV

SPECIFIC HEAT CALCULATION FOR DATA OF TABLE III AND FIGURE 7

| Temp. (K) | $\rho$ (Ohm-cm)         | $I_{\infty}^2 - I_f^2$ (Amps <sup>2</sup> ) | T (T/sec) | $C_p^*$ (J/gmK) |
|-----------|-------------------------|---|-----------|-----------------|
| 1860      | $74.395 \times 10^{-6}$ | 8051.2                                      | 40.6      | 0.167           |
| 1837      | $73.550 \times 10^{-6}$ | 7346.7                                      | 35.5      | 0.172           |
| 1808      | $72.608 \times 10^{-6}$ | 6527.9                                      | 31.5      | 0.170           |

$$^*C_p = \frac{\rho}{A^2 \delta} \frac{(I_{\infty}^2 - I_f^2)}{\dot{T}} \quad \text{where } A^2 \delta = 0.088238 \text{ gm/cm.}$$

#### IV. EXPERIMENTAL RESULTS AND DISCUSSION

Experimental measurements were made on tantalum and tantalum-10 tungsten alloy. These materials were chosen primarily because samples which were used in the apparatus for steady state measurements were on hand. Thus, thermal conductivity, electrical resistivity, Lorenz ratio, total hemispherical emittance at 0.65 microns and Thomson coefficient data were already available <sup>(5)</sup>. Therefore, developing the capability of measuring specific heat on these samples automatically demonstrates the feasibility of calculating thermal diffusivity as well as enthalpy from the data and increases the number of properties measured with the multiproperty apparatus to nine. In addition to the convenience of having specimens available with a suitable geometry, the specific heat of tantalum at moderate temperatures (1000 to 2000 K) is relatively well established and the specific heat of the Ta-10W alloy should be very close to that of pure tantalum.

The tantalum samples are described elsewhere <sup>(5)</sup>. They consist of 1/8 inch outside diameter by 0.040 inch inside diameter thick wall tubes of metallurgical grade material. The nominal composition is 99.9 percent tantalum with small amounts of silicon, magnesium, copper, and calcium and traces of iron, nickel, and zirconium present. The density is 16.593 gm/cm<sup>3</sup>. The Ta-10W alloy is from a batch used in a high temperature reference program. Only a few measurements were made on this material.

The original measurements were made with the pyrometer sighting on the sample surface. It was discovered that such a procedure yielded poor specific heat results when data taken near the start of the transient were used. Similar results were obtained with the Ta-10W alloy sample. There may be several reasons for this behavior. The rate of change of surface temperature just after the initiation of the transient may not be equal to the rate of change of the effective sample temperature for a 1/8 inch or larger diameter rod of tantalum. Also the change in power input is not a step function (Figure 6). While a portion of the data taken immediately after the initiation of the transient was discarded, it is possible that the first part of the data used in the calculations were still influenced by the departure from a step function.

Some measurements were made using the change in surface temperature measured during the middle and near the end of the transient. Specific heat values calculated from these data were markedly improved and some of these data are included in Figure 8 which compares the present results to literature values. The

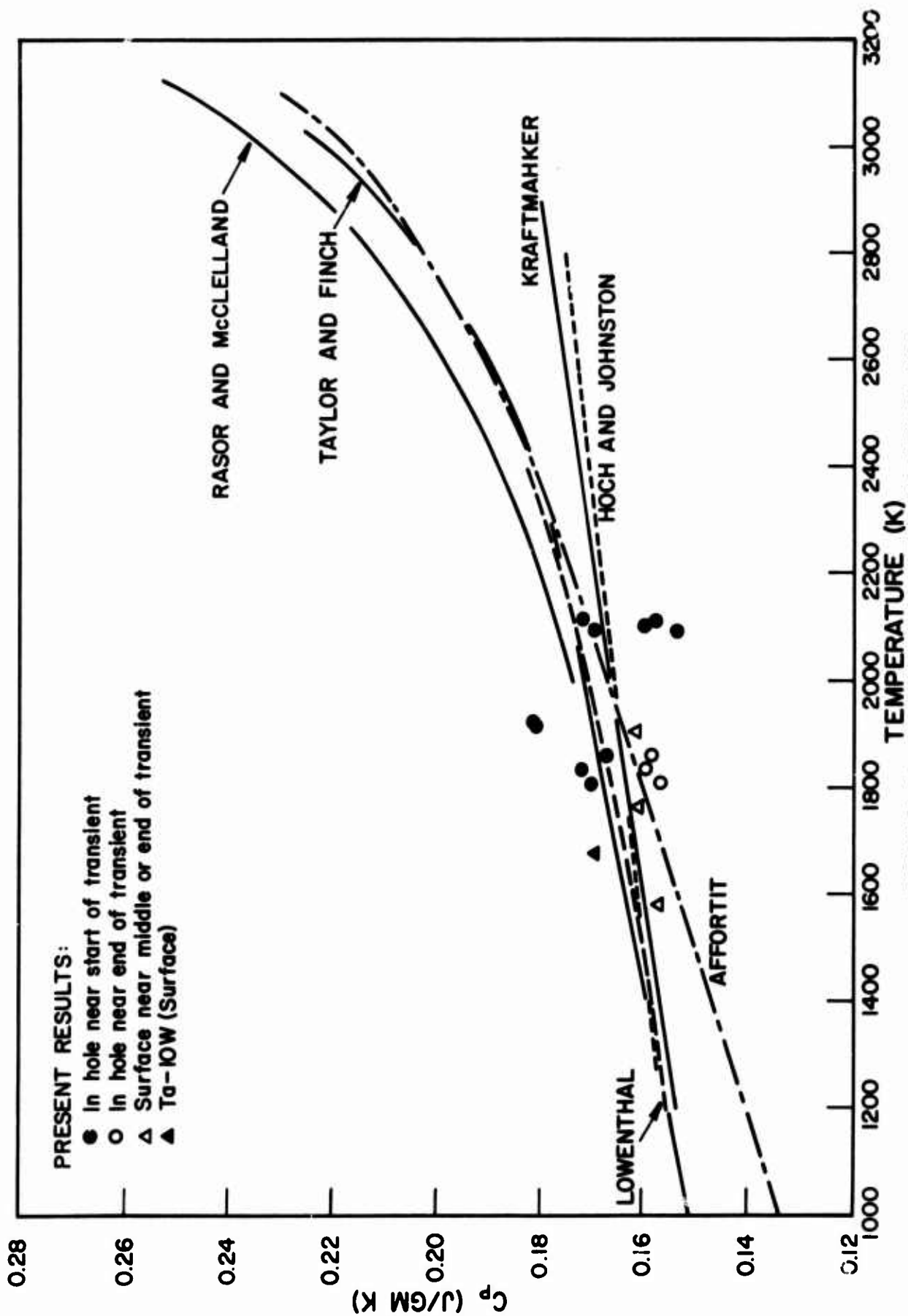


FIGURE 8. SPECIFIC HEAT OF TANTALUM

improvement in these results is believed to be caused primarily by rate of change of surface temperature approaching the rate of change of effective temperature near the end of the transient.

A 0.060 inch diameter hole was drilled perpendicular to the sample axis to intercept the inner bore. The effective emittance of this hole was determined from the brightness temperature and from the electrical resistivity versus temperature curve. Thus temperature measurements of the interior of the sample during temperature transients could be directly measured using the automatic optical pyrometer. Using these measurements, a significant improvement in the specific heat results was noted. Results calculated from measurements near the initiation and near the end of the transient were in much closer agreement than those obtained from surface measurements.

Typical results using temperature measurements in the hole are shown in Figure 8. From this figure it is seen that the present experimental results using the hole are in reasonable agreement with literature values <sup>(10, 11, 13, 14, 15)</sup>, but that the scatter is considerable - up to  $\pm 8$  percent. Cooling rates from 31 to 90 K/sec were used in obtaining these results. Most of the scatter in experimental values is due to the uncertainty in determining  $\dot{T}$  (Eq. 9). This scatter would be greatly reduced with improved data recording instrumentation.

Specifically, if a digital data acquisition system including a small computer for transient data storage (or an auxiliary storage unit) were used, the voltage, current, temperature, and rate of change of resistivity could be recorded during the transient to a much greater degree of accuracy than the present system based on an oscilloscope. Then  $\dot{T}$  could be determined from the rate of change of resistivity ( $\dot{\rho}$ ) and the known temperature coefficient of resistivity, i. e.,  $\dot{T} = \rho / (d\rho/dT)$ . This procedure, which was successfully used by Taylor and Finch <sup>(10)</sup>, has the advantage of determining the rate of change of the effective temperature, rather than the rate of change of the surface temperature. Also problems associated with the temperature dependence of the spectral emittance and window correction and the non-linear pyrometer response are avoided. In addition, the measurement of  $\dot{\rho}$  can be obtained quite accurately using a digital data acquisition system. For the example given in Table III and Figure 7, the change in voltage at 66.940 amps for a temperature change from 1906 to 1774 K is 0.010569 volts. Using a digital data system and an amplifier, this voltage change could be measured to better than 0.1 percent and  $\dot{T}$  could be determined within 1 percent. Thus the specific heat could be determined within  $\pm 1$  percent at high temperatures.

## V. SUMMARY AND CONCLUSIONS

The multiproperty apparatus can be used to determine the specific heat at high temperatures (1000-2800 K) on the same sample used for thermal conductivity, electrical resistivity, total hemispherical emittance, spectral emittance at 0.65 microns, Thomson coefficient, and Lorenz ratio measurements. Enthalpy and thermal diffusivity can thus be calculated from the results. The specific heat determination involves small transients from an established steady-state temperature, and should not put severe strain on the samples nor cause significant internal changes.

The equation used to calculate specific heat from the experimental data is

$$C_p = \frac{\rho}{\Lambda^2 \delta} \frac{(I_f^2 - I^2)}{T}$$

All quantities entering into this equation except  $T$  could be measured to about five significant figures. Consequently, the accuracy of the  $C_p$  determination was controlled by the ability to determine  $T$ . Using an automatic pyrometer and an oscilloscope, this uncertainty was about  $\pm 8$  percent. However, using a commercially available digital data acquisition system, the inaccuracy should be reduced by about an order of magnitude.

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